

2003年 1月29日 16時14分

小島国際特許事務所

NO. 7730 P. 2



0039-7632-0X

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF :

HIROYUKI YANO ET AL : GROUP ART UNIT:1765

SERIAL NO: 09/531,163 :

FILED: MARCH 17,2000 : EXAMINER:DEO,D.

FOR: AQUEOUS DISPERSION, AQUEOUS  
DISPERSION FOR CHEMICAL  
MECHANICAL POLISHING USED  
FOR MANUFACTURE OF SEMI-  
CONDUCTOR DEVICES, METHOD  
FOR MANUFACTURE OF SEMI-  
CONDUCTOR DEVICES, AND  
METHOD FOR FORMATION OF  
EMBEDDED WIRING

DECLARATION UNDER 37 C.F.R. § 1.132

ASSISTANT COMMISSIONER FOR PATENTS  
WASHINGTON, D.C. 20231

SIR:

Now comes Masayuki Hattori, who deposes and states that

1. I am named as an inventor in the above-identified application.
2. I am a graduated of WASEDA University and received my bachelor degree in the year 1982.
3. I have been employed by JSR Corporation since 1982, and I have been conducting research in the field of for 7 years.
4. I am familiar with the prosecution history of U.S Application Serial No.09/531,163 and I have read and understood the contents of the U. S. Patent No.5,876,490 (Ronay) which was cited by the Examiner against the claims of 09/531,163.
5. In order to compare the polishing performance of the dispersions claimed in the above-mentioned U.S. application with the slurries of the Ronay

patent, the following experiments were carried out by me or under my direct supervision and control.

[ EXPERIMENT 1 ]

1. Preparation of aqueous dispersion for polishing.

(1) Slurry by JSR method (as described in Example 2A of the present application)

An aqueous dispersion for polishing is prepared by blending 0.7% of a polymer particle (b) (crosslinked polymethyl methacrylate-based particle having carboxyl group, amide group, ester group on the surface of the particle and 0.17 $\mu$ m in mean particle diameter) and 5% of a fumed alumina particle in an ion-exchanged water. The pH was adjusted to 4 with nitric acid. The zeta potential of the polymer particle (b) was -24 mV, and the zeta potential of the alumina particle was +35 mV.

A dispersed particle in the slurry was observed with a transmission electron microscope, and it was particle that alumina was attached to the surface of polymer (b). An aggregated particle of 1 - 10 $\mu$ m in mean particle diameter was formed generously by the composite particles.

(2) Slurry by Ronay method (comparative experimental example).

An aqueous dispersion for polishing was prepared by blending 1.5% of polyacrylic acid (molecular weight 7000) and 5% of fumed alumina particle into an ion-exchanged water.

The polyacrylic acid is absorbed on the surface of the alumina particle, the alumina particle was dispersed into water made in a finer state. When observing it by a transmission electron microscope, its mean particle diameter was 130 nm.

(3) Comparative example.

A slurry containing 5% of alumina was prepared without using the additive agent (polyacrylic acid) and then the slurry was adjusted to pH 4 with nitric acid. In this case, the mean particle diameter was 150 nm.

2. Method of test

An aluminum film (film thickness 5000 $\text{\AA}$ , containing 1% copper) attached to the thermally oxidized film of 8 inches, as polished in Experimental Example 2C of the present application was polished using the above-described three kinds of slurries under the same conditions. As for the method, a wafer was set in a CMP apparatus (Model "LPG510"; made by

Lapmaster SFT, Corporation), IC1000 was used as a pad, 300g/cm<sup>2</sup> was loaded and the wafer was polished for 1 minute under the conditions of the head/table at the speed of 125 rpm/120rpm and the slurry discharge at 250 ml/min.

### 3. The results of test

By the JSR method, the polishing rate was 3900Å/min and the number of scratches was 30 points or less, hence the results were excellent.

On the other hand, in the Comparative Experiment Example utilizing a slurry according to Ronary method, since alumina was finely dispersed, the polishing rate was 410Å/min, specifically, extremely lowered. Moreover, since there is no suitable cushion effect, the number of the scratches was 200 points or more, scratches were found in the whole of the wafer.

For reference purpose of comparison, A slurry containing 5% of alumina was prepared without using the additive agent and then the slurry was adjusted to pH 4 with nitric acid. In this case, it was found that article diameter was 150 nm, the polishing rate was 1900Å/min and the number of the scratches was 85 points.

4. The above-described experiment provides direct comparison of the polishing rate and the number of scratches presently claimed composition with ( i ) a composition that does not contain a polymer particle and ( ii ) a composition of the Ronay patent. The results of these tests are tabulated below.

	Polishing rate	Number of scratches
Present dispersion Example 2A(Sp/Si=1.3)	3,900Å/min	30pts
( i ) Alumina particles(no polymer particle)	1,900Å/min	85pts
( ii ) Ronay slurry	410Å/min	200pts

5. As described in the "Method of test" above, the polishing rate and the number of scratches were measured under the same conditions for all samples.

6. The tests and methods are art-recognized and produce statistically significant results.

7. The polishing rate of a dispersion adhering to the present claim limitations is shown to be significantly greater than the polishing rate obtained for the prior art composition or for a slurry containing only alumina particles , and the dispersion adhering to the present

claim limitations produces a polished surface having fewer scratches.

8. The polishing rate observed for the presently claimed composition would not be expected from the disclosure of the Ronay specification which discloses an inhibitory effect for polishing rate when a polymer particle and inorganic particle are combined in a polishing slurry.

#### [ EXPERIMENT 2 ]

##### 1. Preparation of aqueous dispersion for polishing.

###### (1) Synthesis of styrene polymer particles (R1)

After dissolving 40g of benzoyl peroxide in 1000g of styrene monomer, this solution was dispersed in 4000g of water containing 40g of sodium lauryl sulfate. Obtained solution was homogenized in a finer state by a high-pressure homogenizer ("Micro-fluidizer" by Mizuho Corporation) at a pressure of 800 kg/cm<sup>2</sup>. Emulsified liquid was polymerized for 8 hours at 85°C while stirring in a 7-liter volume glass-flask. This yielded polystyrene particles had a mean particle size of 0.28µm. We refer to this styrene polymer particles as (R1).

###### (2) Slurry by JSR method

An aqueous dispersion for polishing is prepared by blending 0.7 % of a polymer particle (a) (as described in Synthesis Example 1 of the present application, carboxy-modified polystyrene particle having carboxyl group and sulfuric ester group on the surface of the particle and 0.24 µm in mean particle diameter) and 5% of a fumed alumina particle in an ion-exchanged water. The pH was adjusted to 4 with nitric acid.

###### (3) Slurry by Ronay method (comparative experimental example).

A slurry by Ronay method was prepared by the above-mentioned method using styrene polymer particles (R1) in place of carboxy-modified polystyrene particle (a).

###### (4) Comparative example.

A slurry containing only alumina particles was prepared without using the additive agent.

#### 2. Method of test

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The method of test is the same as the method of test used in experiment 1.

3. The results of test

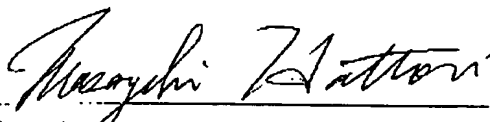
The results of these tests are tabulated below.

	Polishing rate	Number of scratches
Using polymer particle (a);JSR method	3,900Å/min	30pts
Using polymer particle (R1);Ronay method	650Å/min	65pts
Alumina particles(no polymer particle)	1,900Å/min	85pts

As shown in the table, the polishing rate of the slurry consisting carboxy-modified polystyrene particle having carboxyl group and sulfuric ester group on the surface of the particle is shown to be significantly greater than the polishing rate obtained for the slurry consisting simple polystyrene or for a slurry containing only alumina particles, and the slurry consisting carboxy-modified polystyrene particle produces a polished surface having fewer scratches.

4. As described in the "Method of test" above, the polishing rate and the number of scratches were measured under the same conditions for all samples.

5. The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

  
Signature

Jan. 28, 2003  
Date